

TEST PROCEDURE

TP 714C

Title Diesel Particulate Filter Handling and Weighing	Page Number 1 of 17
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Revision Description

- (1) 09-30-94 The purpose of this change is to revise the procedure as described in EPCN #170.

Note: Specific brand names in EPA/EOD procedures are for reference only and are not an endorsement of those products.

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1. Purpose

The purpose of this procedure is to prepare filters used for collecting particulate samples during the light-duty diesel emission test and to measure the particulate sample after collection.

2. Test Article Description

Filters to be placed in a tri-filter assembly. Primary and backup filters are used for each phase (bag) of the Federal Test Procedure (FTP).

3. References

- 3.1 “Code of Federal Regulations;” Title 40, Part 86 Subparts A and B
- 3.2 Cahn C-30/31 Instruction Manual for Cahn C-30 Microbalance
- 3.3 Environmental Protection Agency (EPA) Drawing T0319C, Tri-filter Assembly
- 3.4 Memo, Bill Courtois, December 20, 1990, Subject: “Particulate Test Processing System Modifications”

4. Required Equipment

- 4.1 Cahn model C-30 microbalance (or equivalent) and its instruction manual
- 4.2 Filters for collecting diesel particulate matter (47-mm Pallflex brand, Model T60A20)
- 4.3 Tri-filter assembly, with primary and backup filters for each of three phases
- 4.4 Temperature and humidity recording system to provide a permanent record of the ambient conditions in the filter conditioning room
 - 4.4.1 General Eastern Dew-Point Hygrometer Hygro-M2 for measuring ambient humidity

- 4.4.2 Type “J” thermocouple for measuring ambient temperature
- 4.4.3 Soltec strip chart recorder Model 3316 to record temperature and humidity measurements
- 4.4.4 Macintosh computer and Excel software to calculate relative humidity from temperature, humidity, and barometric pressure readings
- 4.5 Non-magnetic, non-serrated, duckbill forceps for handling the filters and non-magnetic, non-striated, pointed-end forceps for handling the calibration weight
- 4.6 Form 703-02, “Diesel Vehicle Preconditioning,” (see TP 703)
- 4.7 Form 903-01, “Variant Test Report”
- 4.8 Form 714-01, “Particulate Filter Record Data Sheet,” (Attachment A)
- 4.9 Form 714-02, “Particulate Test Data Sheet,” (Attachment B)

5. Precautions

- 5.1 Extreme care must be used in the operation of the microbalance.

Instructions for its proper use are detailed in Sections 7 and 8 of this procedure and in the instruction manual kept in the weighing room.

Technicians must be familiar with the instruction manual before attempting to use the balance.

Contact with the radioactive static eliminator contained within the balance must be avoided.
- 5.2 Skin contact with equipment contaminated with particulate matter must be avoided.
- 5.3 Never handle the filters and the calibration weight with the fingers. Contamination by touching will give erroneous measurements.

If the calibration weight or the filters are accidentally handled with the fingers, contact Vehicle Testing (VT) supervisory personnel.

- 5.4 Non-magnetic, non-striated forceps must be used when handling filters and non-magnetic, non-striated, pointed-end forceps must be used for handling the calibration weight.

6. Visual Inspections

All visual inspections are incorporated as part of the test article preparation and test procedure.

7. Test Article Preparation

Steps covered in this section are performed before the particulate sample is taken. Filters are conditioned and weighed in batches consisting of two to six filters.

A typical three-bag FTP would require a batch of six filters, one primary and one backup filter for each phase.

- 7.1 Observe the temperature and humidity record chart in the weighing room for adherence to the following tolerances:
- 7.1.1 The temperature of the room in which the filters are conditioned and weighed shall be maintained to within 10 °F (6 °C) of a set point between 68 and 86 °F (20-30 °C) during all filter conditioning and weighing.
- The set point is defined as the mean temperature during the time any filters are conditioning in the weighing room.
- To maintain ambient temperature conditions that meet both light-duty and heavy-duty requirements, the ambient temperature shall be maintained between 68 and 77 °F.
- A continuous recording of the temperature is required.
- 7.1.2 The relative humidity (RH) of the room shall be maintained to within 10 percent of a set point between 30 and 70 percent during all filter conditioning and weighing.

- 7.1.3 The set point is defined as the mean humidity during the time any filters are conditioning in the weighing room.

To maintain ambient humidity conditions that meet both light duty and heavy duty requirements, the dew point shall be maintained between 45.5 and 52.7 °F, which will result in a relative humidity range of 38.67 to 50.62 percent at 72 °F ambient temperature and 29.00 Hg barometric pressure.

A continuous recording of the dew point is required.

- 7.2 When the vehicle to be tested has completed the LA-4 preconditioning cycle (TP 703), begin the soak period for filters to be used for collecting particulate samples:

- 7.2.1 Mark six clean petri dishes with consecutive filter numbers (taken from the log sheet posted on the weighing room door) and the test number.

- 7.2.2 On Forms 714-01 and 703-02 and the ambient strip chart recorder, enter the soak start time, date, and filter numbers for the test.

- 7.2.3 Label the temperature recording chart with the strip chart recorder equipment tracking (ET) ID number and date.

- 7.2.4 Using the duckbill forceps, carefully place a filter in each petri dish and partially cover it with the lid.

Ensure that the opening created by the placement of the lid is not over the filter.

This will allow the filter to get the proper ventilation and reduce the chance of dust collecting on the filter during conditioning.

- 7.2.5 Allow the filters to condition in the same dishes for 8-56 hours before weighing.

Verify that the ambient temperature and humidity conditions adhere to the tolerances given in Steps 7.1.1 and 7.1.2.

7.3 Calibrate the microbalance.

Before using the microbalance, allow a 2-hour warm-up period if it has been turned off. Standard operating procedure dictates that the balance be left on continuously for maximum stability and precision.

If the microbalance has been turned off, determine the reason to see if there is cause for it to remain turned off.

This balance incorporates the use of a pan brake. The use of the pan brake is optional. The pan brake holds the balance pans steady when the calibration weight or filters are placed on or off the pan.

When zeroing, calibrating, or weighing a filter, ensure that the sample pans are not moving and that the weight display (LED) is stable.

It may take 30 seconds or more for the display to stabilize after placing the calibration weight or filter on the sample pan.

7.3.1 Engage the brake (raise the heads) by pressing the “Brake” button.

7.3.2 After placing the calibration weight or filter on the pan, release the brake (lower the heads) by pushing the “Brake” button again.

The balance is now ready for the weighing process.

7.3.3 Ensure that the “A” 200-mg weighing range is selected.

If not, press the “RANGE” button to select the “A” range.

7.3.4 Zero the microbalance by pressing the “TARE” button. The weight display should read 0.000 mg.

If not, press the “TARE” button again.

Repeat this procedure as necessary until the display stays at 0.000 mg.

7.3.5 Place the 200-mg Class 1.1 calibration weight on the sample (left) pan using the non-magnetic, non-striated, pointed-end forceps.

- 7.3.6 When the sample pans are not moving and the weight display has stabilized, push the “CAL” button once. The weight display should read 200.000 mg.
- If “E3” appears on the weight display, the calibration weight does not match the weighing range.
- If the weight display changes from 200.000 mg, push the “CAL” button again.
- Repeat this procedure until the weight display stays at 200.000 mg.
- 7.3.7 Remove the 200-mg calibration weight from the sample pan and return it to its container.
- 7.3.8 Ensure that the weight display returns to 0.000 mg.
- If not, repeat Steps 7.3.2 through 7.3.4 until the display returns to 0.000 mg after calibration.
- 7.4 Weigh the two reference filters. These filters must remain in the weighing room at all times.
- 7.4.1 Place one reference filter on the sample (left) pan, with the range selector in the 200-mg position, and obtain a stable reading.
- 7.4.2 Record the required information under the “Reference Filter Data” section of Form 714-01.
- 7.4.3 Calculate the relative humidity from the ambient temperature and dew-point temperature using the relative humidity calculator on the Macintosh computer.
- 7.4.4 Remove the filter from the balance and replace it in its petri dish.
- 7.4.5 Repeat Steps 7.4.1 through 7.4.4 using the second reference filter.
- 7.4.6 If the weight of either of the reference filters changes between pre- and post-test sample filter weighing by more than ± 2.0 percent of the test average primary filter loading (recommended minimum of 0.5 mg) or ± 0.010 mg, whichever is greater, then the post-test sample filter weights are invalid.
- The post-test weighing procedure can be repeated to obtain valid weights within the time limits as specified in 40 CFR 86.139.

- 7.4.6 During diesel testing programs, reference filters shall be changed at least once a month.
- 7.5 Record the required information under the “Ambient Conditions Log” section of Form 714-01.
- 7.6 Pre-test filter weighing (tare weight):
- 7.6.1 Using the duckbill forceps, place a test filter on the sample (left) pan and obtain a stable reading.
- 7.6.2 Record the required information under the “Test Filter Data” section on Form 714-01.
- 7.6.3 Remove the filter from the balance and return it to its petri dish.
- 7.6.4 Repeat this process for all six test filters.
- 7.6.5 On Form 703-02, record the filter numbering range and the time the filters were weighed as the “Pre-Test soak start time.”
- 7.7 Using the duckbill forceps, carefully place the filters in the tri-filter assembly.
- The tri-filter assembly consists of an adapter with a single inlet tube and three branch tubes, each with two filter holders.
- 7.7.1 Position the filtering apparatus with the single tube down and open the filter holder for Filter #1. Using the duckbill forceps, remove the metal backup plate.
- 7.7.2 Remove any residual particulate matter from the assembly with a lint-free disposable wiper.
- 7.7.3 Using the duckbill forceps, carefully insert the filter disc, loading side down, and the metal backup plate.
- Tighten the filter holder.
- 7.7.4 Open Filter Holder #2 and repeat Steps 7.7.1 through 7.7.3 for Filter #2, which is the backup filter for Filter #1.
- 7.7.5 Repeat the entire sequence (Steps 7.7.1 through 7.7.4) for the remaining tri-filter assembly branch tubes, inserting Filter Pairs #3 and #4 and #5 and #6.

7.8 On Form 703-02 and the ambient strip chart, enter the time the filter assembly is removed from the weighing room. Also record the equipment tracking identification number of the strip chart recorder.

7.9 The particulate test must begin within one hour after the loaded tri-filter assembly is removed from the weighing room, or within eight hours if:

(1) the tri-filter assembly ends are kept plugged

or

(2) the tri-filter assembly is attached to the particulate sampling system, as long as no flow occurs across the filters.

8. Test Procedure

This section covers all steps performed during and after the emission test that are not covered in TP 713.

101 Attach the tri-filter assembly to the particulate probe and collect the particulate sample according to the instructions outlined in TP 713.

102 When the test is completed, disconnect the vacuum lines from the filter assembly. Remove the assembly from the sample probe line.

103 As soon as possible, and in no case longer than one hour after the test, return the assembly to the weighing room and carefully place it in its holder.

104 Separate Filter Holder #2 from the first branch of the assembly and remove the metal backup plate.

105 Using the duckbill forceps, carefully remove the backup filter and place it in Petri Dish #2 with the contaminated side facing up.

Check the filter to ensure that small pieces of the filter edge did not tear off while in the assembly. If the filter appears to have been damaged, notify the team leader.

Partially cover the dish.

- 106 Remove the primary filter from the first branch of the assembly, following the instructions given in Steps 104 and 105.

The particulate will be on the bottom of the filter for Filter #1.

Turn it over and place it in Petri Dish #1 without touching or otherwise disturbing the particulate collected on the filter.

Should the sample on the filter contact the petri dish or any other surface, the test is void and must be rerun.

- 107 Repeat Steps 104 through 106 for the filters in “Branch 2” of the assembly.

- 108 Repeat Steps 104 through 106 for the filters in “Branch 3” of the assembly.

- 109 Record the start of post-test soak start time on Form 703-02 and the ambient strip chart. Post-test soak time is considered to be the time the filter was placed in the petri dish.

- 110 Allow the filters to condition for 1-56 hours.

- 111 Calibrate the microbalance according to the instructions given in Step 7.3

- 112 Weigh the reference filters (post-test weight) according to the instructions given in Step 7.4.

- 113 Post-test filter weighing (gross weight): Weigh each test filter, according to the instructions given in Step 7.6.

The difference between the post-test (gross) weight and the pre-test (tare) weight is the net weight of the particulate collected on each filter.

- 114 Record the end of the post-test soak time on Form 703-02 and on the ambient strip chart.

- 115 Submit the particulate weighing data to the person doing the processing, where it will be verified for accuracy and sent to computer operations to be processed.

9. Data Input

- 9.1 In the log book kept in the Vehicle Testing team office, record the filter data (reference and test) and ambient conditions of the weighing room.
- 9.2 On Form 714-02, record the dry gas meter data at the vehicle test site (as part of TP 713).
- 9.3 On Form 713-02, record the particulate filter conditioning times.
- 9.4 Label the strip charts with the date and strip chart recorder equipment tracking ID number.

10. Data Analysis

- 10.1 Form 714-01 is submitted to the person processing the data at the completion of the emission test, where it is reviewed for accuracy and completeness.
- 10.2 After review, Form 714-01 is submitted daily to the Computer Room for entry into the various programs which comprise the total particulate test.
- 10.3 Continuous temperature and humidity records are kept in the conditioning room and are available for review upon request.

11. Data Output

- 11.1 All forms and test records are verified by a qualified technician who did not record the data. The verifying technician checks the data for completeness, accuracy, and compliance with EPA regulations.

The verifying technician will write his/her identification number and date in the "Verified By" area of the forms.

On the strip chart, the technician will write his/her identification number and "OK."

This certifies that the data are accurate and complete.

- 11.2 The particulate filter log sheet is reviewed for adherence to reference filter specifications, ambient temperature and humidity specifications, correct data entries for the test filter pre- and post-test weights, and the reference filter weights.

- 11.3 The tolerance allowed for the reference filters for a particular test must be determined using one of these two methods:

11.3.1 Method 1:

If the weight of either of the reference filters changes between the pre-test and the post-test weighing by less than ± 0.010 mg, all filter weights are valid.

If this tolerance is exceeded, Method 2 must be used to determine if the filter weights are valid.

11.3.2 Method 2:

If the weight of either of the reference filters changes between the pre-test and post-test weighing by more than ± 2.0 percent of the average primary filter loading, the post-test sample filter weights are not valid.

To use this method the nominal filter loading for each of the 3 primary filters must be known.

Calculate the reference filter tolerance using the following example

Example:	<u>Phase</u>	<u>Primary Filter Loading</u>
	1.	0.000577 grams
	2.	0.000569 grams
	3.	<u>0.000543</u> grams
	total	0.001689 grams

“Average primary filter loading”: $0.001689/3 = 0.000563$ grams

“Tolerance for the reference filter weights”:

$$0.000563 \text{ grams} \times .02 = 0.000011 \text{ grams or } 11 \text{ micrograms.}$$

If the calculated tolerance is greater than the change in each of the reference filters then the filter weights are valid.

If they are not, the post test weights are not valid, however, the post-test weighing procedure can be repeated within 1 to 56 hours.

Return to Step 110 and complete the procedure from this point.

12. Acceptance Criteria

Acceptance criteria are based on Federal Register requirements and must be met for the test to be valid.

- 12.1 The filter conditioning room temperature must remain stable to within 10 °F of a set point between 68-86 °F during all filter conditioning and weighing.

The set point is defined as the mean temperature during the time any filters are conditioning in the weighing room.

- 12.2 The filter conditioning room humidity (RH) must remain stable to within 10 percent RH of a set point between 30-70 percent RH during all filter conditioning and weighing.

The set point is defined as the mean humidity during the time any filters are conditioning in the weighing room.

- 12.3 The pre-test filter stabilization period must be from 8-56 hours in length.

- 12.4 The post-test filter stabilization period must be from 1-56 hours in length.

- 12.5 If the weight of either of the reference filters changes between pre- and post-test sample filter weighing by more than ± 2.0 percent of the test average primary filter loading (recommended minimum of 0.5 mg) or ± 0.010 milligrams, whichever is greater, then the post-test sample filter weights are invalid.

- 12.6 Particulate collection must start within one hour after the filters leave the conditioning room, or within 8 hours if:

(1) the tri-filter assembly ends are kept plugged

or

(2) the tri-filter assembly is attached to the particulate sampling system, as long as no flow occurs across the filters.

13. Quality Control Provisions

These provisions are in-house quality checks and do not necessarily require that a test be voided.

- 13.1 The efficiencies of all filters used in a test should agree with each other within 10 percent.

- 13.2 The loaded filter should contain no discernible particles, which may indicate contamination by foreign substances.
- 13.3 Filters whose efficiencies are lower than 80 percent will be flagged in data reduction, and should be inspected for possible damage.
- 13.4 Any unusual fluctuation in the weighing room temperature or humidity should be reported to the team leader (even if it is within the acceptable tolerance limits).
- 13.5 The technician's identification number must appear on all forms and test records, certifying that the data are accurate and complete.
- 13.6 Particulate loading for a 47-mm diameter filter should be a minimum of 0.5 mg. Equivalent loading is recommended for larger diameter filters.

PARTICULATE FILTER DATA

[illegible]

PARTICULATE TEST DATA SHEET

Figure 1 is a schematic diagram of the experimental setup for the study of the effect of the initial concentration of the solution on the rate of crystallization. The diagram shows a series of test tubes arranged in a rack, labeled 1 through 10. Each tube contains a solution and a seed crystal. The tubes are connected by a network of lines representing the flow of the solution. Labels include 'PRIMARY POLYMER', 'SECONDARY POLYMER', 'THERMAL ANALYSIS', 'CRYSTALLIZATION', 'SEED CRYSTAL', 'SOLUTION', 'THERMAL ANALYSIS', 'CRYSTALLIZATION', 'SEED CRYSTAL', 'SOLUTION', 'THERMAL ANALYSIS', 'CRYSTALLIZATION', 'SEED CRYSTAL', 'SOLUTION'. A legend at the bottom left defines the symbols used in the diagram.